# Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US05/005088

International filing date: 16 February 2005 (16.02.2005)

Document type: Certified copy of priority document

Document details: Country/Office: US
Number: 60/545,772
Filing date: 19 February 2004 (19.02.2004)

Date of receipt at the International Bureau: 07 April 2005 (07.04.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)

World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

1302266

# THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

*March 31, 2005*

**THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.**

**APPLICATION NUMBER:** ***60/545,772***
**FILING DATE:** ***February 19, 2004***
**RELATED PCT APPLICATION NUMBER:** ***PCT/US05/05088***

UNITED STATES PATENT AND TRADEMARK OFFICE
DEPARTMENT OF COMMERCE

Certified by

Jon W. Dudas

Under Secretary of Commerce
for Intellectual Property
and Director of the United States
Patent and Trademark Office

021904 16569 U.S. PTO

22856 U.S. PTO 60/545772 021904

PTO/SB/16 (08/03)
Approved for use through 07/31/2006. OMB 0651-0032
U.S Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

# PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c)

**Express Mail Label No.: EL 993859814 US**

| INVENTOR(S) | | |
|---|---|---|
| Given Name (first and middle [if any]) | Family Name or Surname | Residence (City and either State or Foreign Country) |
| Paul | Kohl | Atlanta, GA |
| Christopher W. | Moore | Atlanta, GA |
| Jun | Li | Atlanta, GA |
| Justin | Tullis | Stone Mountain, GA |

☐ *Additional Inventors are being named on the separately numbered sheets attached hereto.*

**TITLE OF THE INVENTION (500 characters max)**

**THIN-FILM MEMBRANES FOR FUEL CELLS**

**CORRESPONDENCE ADDRESS**

*Direct all correspondence to:*

☐ Customer Number: 24504

*OR*

| | | | | | |
|---|---|---|---|---|---|
| NAME | Christopher B. Linder, Ph.D.<br>Thomas, Kayden, Horstemeyer & Risley, L.L.P. | | | | |
| ADDRESS | 100 Galleria Parkway<br>Suite 1750 | | | | |
| CITY | Atlanta | STATE | Georgia | ZIP CODE | 30339-5948 |
| COUNTRY | U.S.A. | TELEPHONE | 770-933-9500 | FAX | 770-951-0933 |

**ENCLOSED APPLICATION PARTS (*check all that apply*)**

☒ Specification *Number of Pages* 25

☐ CD(s), Number

☐ Drawing(s) *Number of Pages* ______

☐ Other (Specify)

☐ Application Data Sheet. See 37 CFR 1.76.

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT

☒ Applicant claims small entity status. See 37 CFR 1.27

☒ A check or money order is enclosed to cover the filing fees

☐ The commissioner is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number

☐ Payment by credit card. Form PTO-2038 is attached.

FILING FEE AMOUNT ($) 80.00

This invention was made by an agency of the United States government or under a contract with an agency of the United States Government.

☐ No.

☐ Yes, the name of the U.S. Government agency and the Government contact number are:

*Respectfully submitted,*

SIGNATURE: [signature]

Date: 2/19/04

REGISTRATION NO.: **47,751**

TYPE or PRINTED NAME: **Christopher B. Linder, Ph.D.**

DOCKET NO.: **62004-8770**

TELEPHONE: (770) 933-9500

***USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT***

This collection of information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 USC 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, Patent and Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450 DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop Provisional Patent Application, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

*If you need assistant in completing the form, call 1-800-PTO-9199 and selection option 2.*

The PTO did not receive the following listed item(s) page 21 (total = 24 pages)

PTO/SB/17 (10-03)
Approved for use through 10/31/2002. OMB 0651-0032
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

021904 U.S. PTO 16588

# FEE TRANSMITTAL for FY 2004

*Effective 10/01/2003. Patent fees are subject to annual revisions.*

☒ Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT ($) 80.00

| Complete If Known | |
|---|---|
| Application Number | TBA |
| Filing Date | February 19, 2004 |
| First Named Inventor | Kohl, et al. |
| Examiner Name | TBA |
| Group / Art Unit | TBA |
| Attorney Docket No. | 62004-8770 |

## METHOD OF PAYMENT (*check all that apply*)

☒ Check ☐ Credit Card ☐ Money Order ☐ Other ☐ None

☒ Deposit Account

Deposit Account Number: **20-0778**

Deposit Account Name: **Thomas, Kayden, Horstemeyer Risley, L.L.P.**

**The Commissioner is authorized to:** (*check all that apply*)

☐ Charge fee(s) indicated below ☒ Credit any overpayments

☒ Charge any additional fee(s) during the pendency of this application

☐ **Charge fee(s) indicated below, except for the filing fee to the above-identified deposit account**

## FEE CALCULATION

### 1. BASIC FILING FEE

| Large Entity Fee Code | Large Entity Fee ($) | Small Entity Fee Code | Small Entity Fee ($) | Fee Description | Fee Paid |
|---|---|---|---|---|---|
| 1001 | 770 | 2001 | 385 | Utility Filing Fee | |
| 1002 | 340 | 2002 | 170 | Design Filing Fee | |
| 1003 | 530 | 2003 | 265 | Plant Filing Fee | |
| 1004 | 770 | 2004 | 385 | Reissue Filing Fee | |
| 1005 | 160 | 2005 | 80 | Provisional Filing Fee | 80.00 |
| | | | | **SUBTOTAL (1)** | ($)**80.00** |

### 2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE

| | | | Extra Claims | | Fee From Below | | Fee Paid |
|---|---|---|---|---|---|---|---|
| Total Claims | | - 20**= | | X | 9.00 | = | |
| Independent Claims | | - 3** = | | X | 43.00 | = | |
| Multiple Dependent | | | | | 145.00 | = | |

| Large Entity Fee Code | Large Entity Fee ($) | Small Entity Fee Code | Small Entity Fee ($) | Fee Description |
|---|---|---|---|---|
| 1202 | 18 | 2202 | 9 | Claims in excess of 20 |
| 1201 | 86 | 2201 | 43 | Independent claims in excess of 3 |
| 1203 | 290 | 2203 | 145 | Multiple dependent claim, if not paid |
| 1204 | 86 | 2204 | 43 | **Reissue independent claims over original patent |
| 1205 | 18 | 2205 | 9 | **Reissue claims in excess of 20 and over original patent |

**SUBTOTAL (2)** ($)

***or number previously paid, if greater; For Reissues, see above*

## FEES CALCULATION (*continued*)

### 3. ADDITIONAL FEES

| Large Entity Fee Code | Large Entity Fee ($) | Small Entity Fee Code | Small Entity Fee ($) | Fee Description | Fee Paid |
|---|---|---|---|---|---|
| 1051 | 130 | 2051 | 65 | Surcharge-late filing fee or oath | |
| 1052 | 50 | 2052 | 25 | Surcharge-late provisional filing fee or cover sheet | |
| 1053 | 130 | 1053 | 130 | Non-English specification | |
| 1812 | 2,520 | 1812 | 2,520 | For filing a request for *ex parte* reexamination | |
| 1804 | 920* | 1804 | 920* | Requesting publication of SIR prior to Examiner action | |
| 1805 | 1,840* | 1805 | 1,840* | Requesting publication of SIR after Examiner action | |
| 1251 | 110 | 2251 | 55 | Extension for reply within first month | |
| 1252 | 420 | 2252 | 210 | Extension for reply within second month | |
| 1253 | 950 | 2253 | 475 | Extension for reply within third month | |
| 1254 | 1,480 | 2254 | 740 | Extension for reply within fourth month | |
| 1255 | 2,010 | 2255 | 1,005 | Extension for reply within fifth month | |
| 1401 | 330 | 2401 | 165 | Notice of Appeal | |
| 1402 | 330 | 2402 | 165 | Filing a brief in support of an appeal | |
| 1403 | 290 | 2403 | 145 | Request for oral hearing | |
| 1451 | 1,510 | 1451 | 1,510 | Petition to institute a public use proceeding | |
| 1452 | 110 | 2452 | 55 | Petition to revive-unavoidable | |
| 1453 | 1,330 | 2453 | 655 | Petition to revive-unintentional | |
| 1501 | 1,330 | 2501 | 665 | Utility issue fee (or reissue) | |
| 1502 | 480 | 2502 | 240 | Design issue fee | |
| 1503 | 640 | 2503 | 320 | Plant issue fee | |
| 1460 | 130 | 1460 | 130 | Petitions to the Commissioner | |
| 1807 | 50 | 1807 | 50 | Processing fee for provisional application | |
| 1806 | 180 | 1806 | 180 | Submission of Information Disclosure Stmt | |
| 8021 | 40 | 8021 | 40 | Recording each patent assignment per property (times number of properties) | |
| 1809 | 770 | 2809 | 385 | Filing a submission after final rejection (37 CFR § 1.129(a)) | |
| 1810 | 770 | 2810 | 385 | For each add. invention to be examined (37 CFR 1.129(b)) | |
| 1801 | 770 | 2801 | 385 | Request for Continued Examination (RCE) | |
| 1802 | 900 | 1802 | 900 | Request for expedited exam. of a design application | |

Other fee (specify)

**SUBTOTAL (3)** ($)

*Reduced by Basic Filing Fee Paid

| SUBMITTED BY | | | Complete (if applicable) | |
|---|---|---|---|---|
| Typed or Printed Name | Christopher B. Linder, Ph.D. | Registration No. **47,751** | Telephone Number | 770-933-9500 |
| Signature | [signature] | | Date 2/19/04 | |

**WARNING: Information on this form may become public. Credit Card Information should not be included on this form. Provide credit card information and authorization on PTO-2038**

This collection of information is required by 37 CFR 1.17 and 1.27. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 37 USC 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, Patent and Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450 DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

**PATENTS**
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Kohl, et al.

For: Thin-Film Membranes for Fuel Cells

**CERTIFICATE OF EXPRESS MAIL
FOR PROVISIONAL APPLICATION**

Mail Stop Provisional Patent Application
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir:

Enclosed for filing in the above case are the following documents:

Provisional Application Patent Cover Sheet (1 Page)
Provisional Application Consisting of:
25 Page(s) of Specification
Fee Transmittal Form
Provisional Application Filing Fee - $80.00
Return Postcard

Further, the Commissioner is authorized to charge Deposit Account No. 20-0778 for any additional fees required. The Commissioner is requested to credit any excess fee paid to Deposit Account No. 20-0778.

Respectfully submitted,

**Christopher B. Linder, Reg. No. 47,751**

**THOMAS, KAYDEN, HORSTEMEYER & RISLEY**
100 Galleria Parkway, N.W.
Suite 1750
Atlanta, Georgia 30339-5948

Our Reference No: **62004-8770**

---

I hereby certify that all correspondences listed above are being deposited for delivery to the above addressee, with the United States Postal Service **"EXPRESS MAIL POST OFFICE TO ADDRESSEE"** service under 37 CFR §1.10 on the date indicated below.

The envelope has been given U.S. Postal Service "Express Mail Post Office To Addressee" Package **# EL 993859814 US**.

2/19/04
Date

Jo Bryan

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

# BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

## IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.**

Thin-film Membranes for Fuel Cells
Disclosure Summary

Thin-film membranes for use in proton exchange membrane (PEM) fuel cells have been developed. Also included in the development are ways in which to support these films. These membranes are made through common micro-fabrication techniques, including spin-coating and plasma enhanced chemical vapor deposition (PECVD), and can have a thickness up to 20 micrometers. The materials include polymers, silicon dioxide, and doped silicon dioxide. PEM fuel cells membranes work by conducting protons from the cell anode to cathode. The thinner the membrane, the easier it is for protons to move through it, thus increasing the amount of electrical current that can be generated. Meanwhile, these thin-film materials are superior to currently used PEMs in preventing reactants from passing through the membrane, a common problem particularly in direct methanol fuel cells.

One expected use of these thin-film membranes is in micro fabricated fuel cells. The films would be deposited directly onto the substrate during the fabrication sequence. One example of this is in a previous disclosure for micro fabricated fuel cells where the membrane is deposited onto a patterned sacrificial material, which when removed leaves microchannels for fuel flow.

In other applications, the delicate nature of such a thin material requires that it be supported. The support structures include anything with small holes or porous materials, such as fritted glass or a gas diffusion layer. A filler material, such as wax or polymer, is used to fill in the holes. It is then polished to expose the support structure. The membrane is then deposited on the smooth surface. Removal of the filler material leaves the supported membrane with exposed areas for contact between the membrane and reactants. Alternately, the use of a filler material with a high permeability to the reactants would not require its removal.

The fuel cell catalyst can be deposited with these membranes in a variety of methods, including through inks or sputtering. The deposition can be onto or into the support, in between the filler and membrane, or even imbedded into a non-removed filler.

Figure 1 shows a schematic diagram of a supported membrane in its testing setup. Figures 2 and 3 show the polarization curve for a 3-um thick $SiO_2$ film deposited onto a gas diffusion layer with a platinum catalyst loading of 0.35 $mg/cm^2$.

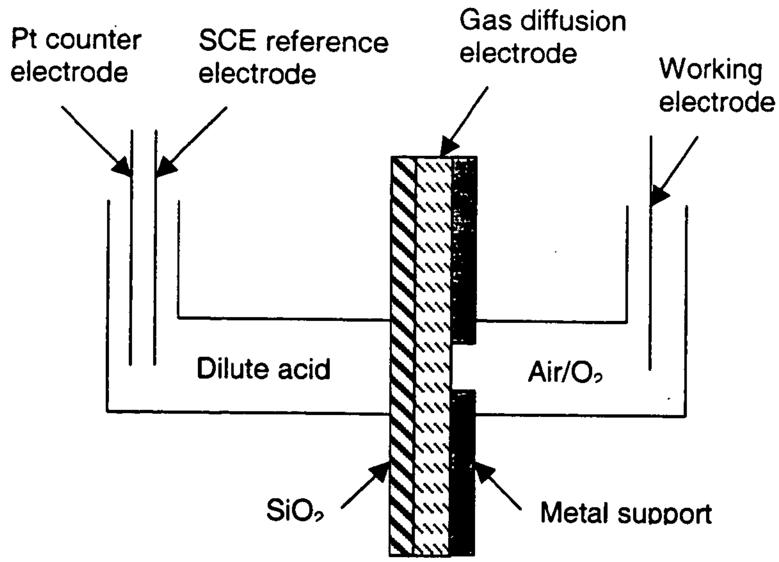



Figure 1: Thin-film membrane support and testing setup

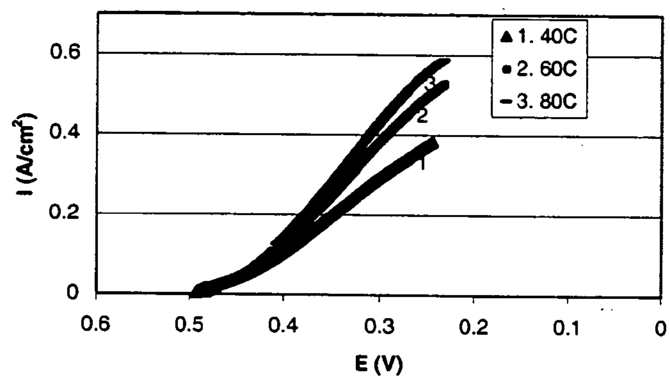



Figure 2: Cathode (air/$O_2$ half cell) polarization performance

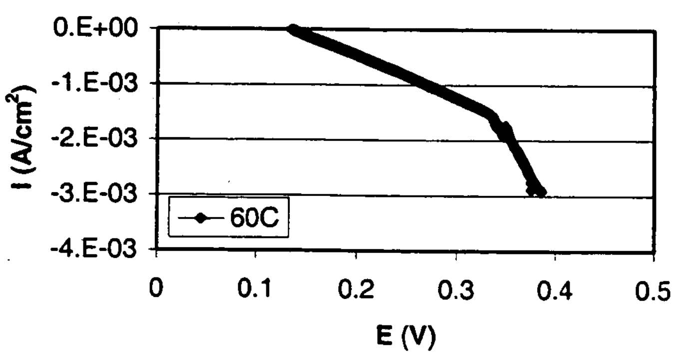



Figure 3: Anode (2M methanol) polarization performance

Thin-film Membranes for Fuel Cells

The following is the first written description of using plasma-deposited $SiO_2$ membranes in microfabricated fuel cells. The concept has been expanded to include other thin-film materials, including doped $SiO_2$, and other fuel cell uses.

Plasma-deposited silicon dioxide was used as an overcoat material. The advantage of $SiO_2$ membranes compared to more traditional materials is the thickness. Table 1.1 below shows a comparison of possible membrane materials for use in thin film fuel cells. Nafion has a higher conductivity, but the films used are much thicker than plasma-deposited $SiO_2$ layers that can be less than 1 $\mu$m. This is important because the resistance (R) of the membrane is related to both its resistivity and thickness. $R = (\rho t)/A$, where $\rho$ is resistivity, t is the thickness, and A is the area. Thus, an important figure of merit for comparing different membranes is the product $\rho t$. While Nafion's resistivity is lower than the low-temperature-deposited $SiO_2$, the thinner $SiO_2$ films should give similar resistances.

Table 1: Fuel cell membrane resistances

| Material | Resistivity ($\Omega$-cm) | Thickness ($\mu$m) | $\rho t = RA$ ($\Omega$-cm$^2$) |
|---|---|---|---|
| Nafion | 100 | 100 | 1.0 |
| High temp. $SiO_2$ (1) | $10^6$ | 0.5 | 50 |
| Low temp. $SiO_2$ | 10,000 | 0.5 | 0.5 |

# Thin-Film Membrane Materials for Use in Microfabricated Direct Methanol Fuel Cells

Christopher Moore, Jun Li, and Paul Kohl

Georgia Institute of Technology

School of Chemical and Bimolecular Engineering

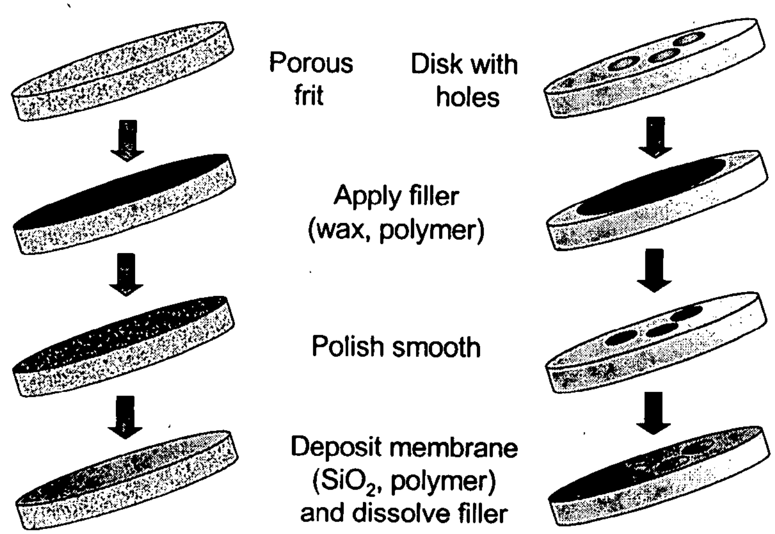
J

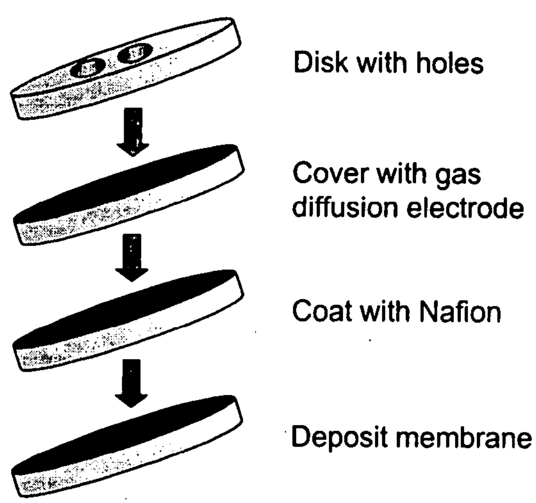
# Introduction

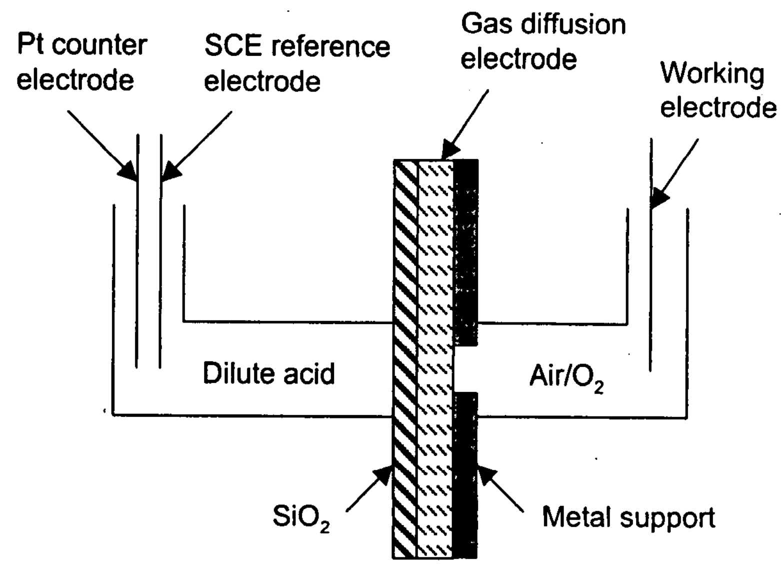
- Motivation
  - Fuel cells offer higher energy density power sources for portable electronics
  - Methanol is conveniently stored in liquid form
  - Improvements must be made to reduce methanol crossover for more concentrated fuel
- Current Work
  - Microfabricated direct methanol fuel cells
  - Integrated on silicon wafer with integrated circuits
- Desired Characteristics of Membranes
  - Thickness: 0.1 – 10 μm
  - Conductivity: 0.01 S/cm
  - Extremely low methanol crossover
  - Good performance at low relative humidity

5

# Membrane Support and Testing




9

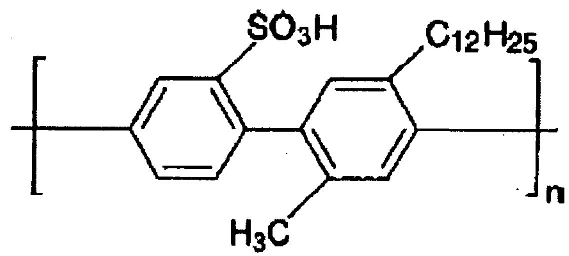
# Catalytic Electrodes

- Porous layer of sputtered platinum-ruthenium at both the anode and the cathode
  - Contact between reactants, catalyst, and membrane
  - Enough platinum to be electrically conductive
- Advantages
  - Lower Pt loading
    - O'Hayre, et al. J. Power Sources **109** (2002) 483-493
  - Reduction of methanol crossover
    - Choi, et al. J. Power Sources **96** (2001) 411-414

7

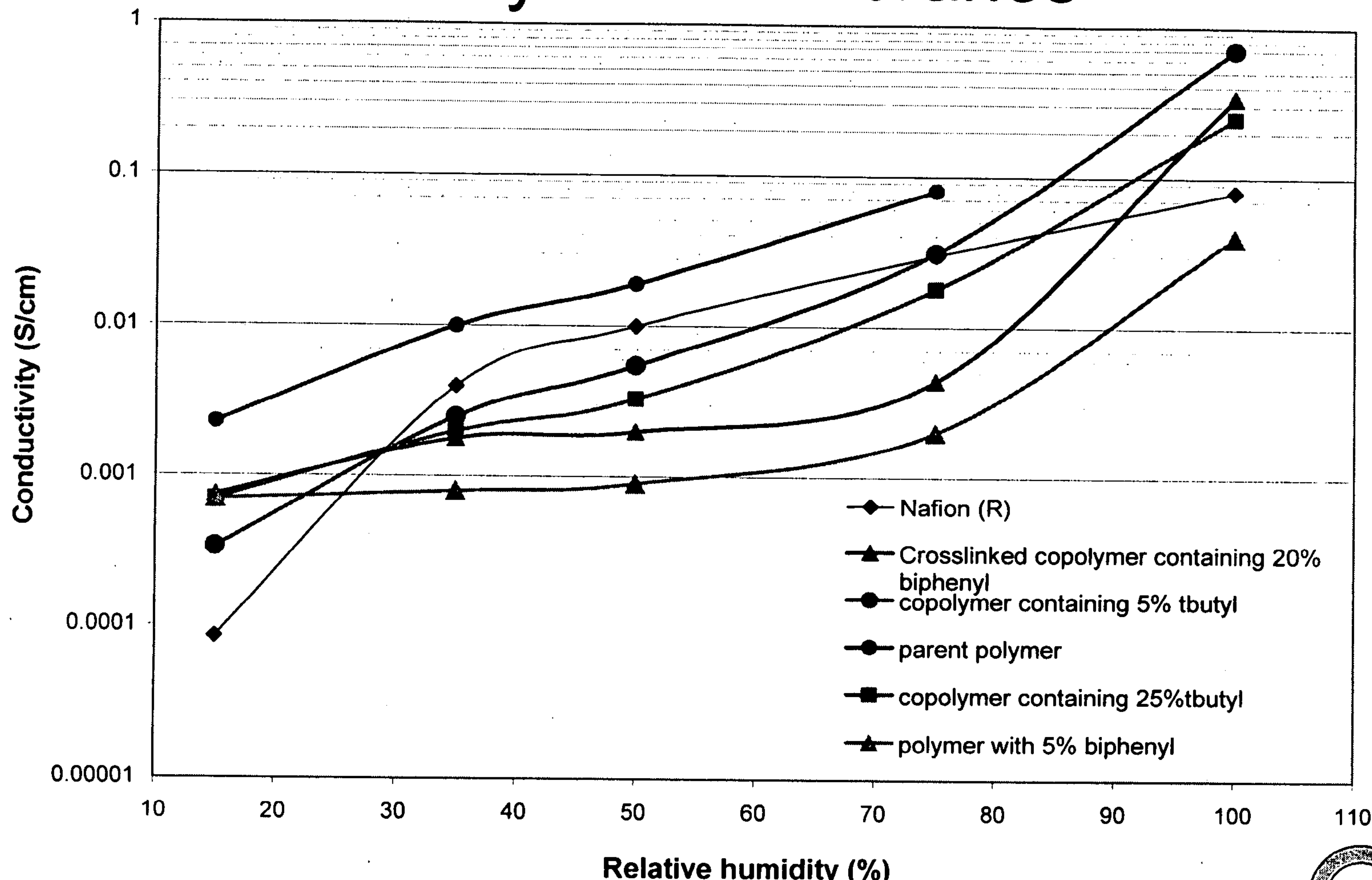
# Polyphenylene Sulfonic Acid

- Received from Case Western Reserve University
  - Dr. Morton Litt and Sergio Granados-Focil




- Soluble in water and methanol
- Needs to be crosslinked for mechanical stability

# Proton Conductivity of Water Insoluble Polymer Membranes

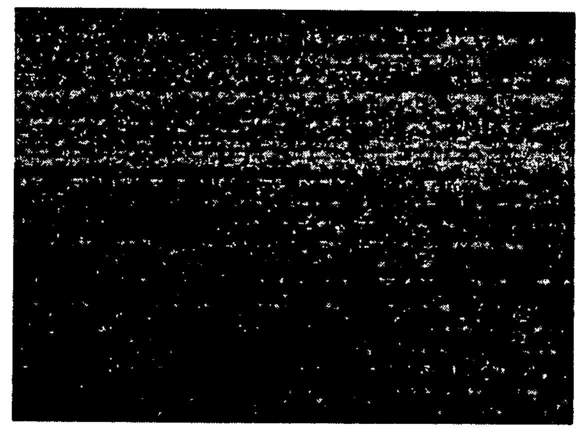



(b)

# Polyphenylene Sulfonic Acid Crosslinking

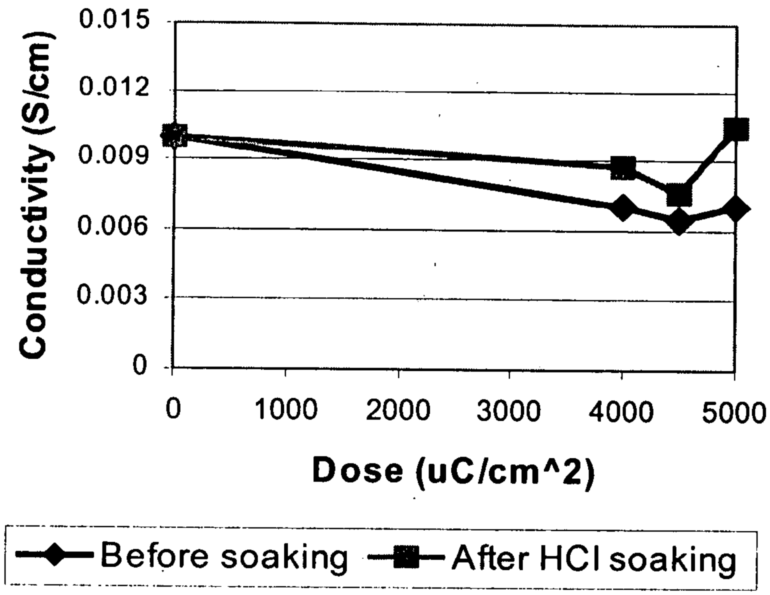
- PPSA films spin coated to thickness of 1 $\mu m$
- Electron beam exposure at 100°C
- No significant change in PPSA thickness
- PPSA film no longer soluble in water or methanol

Before crosslinking

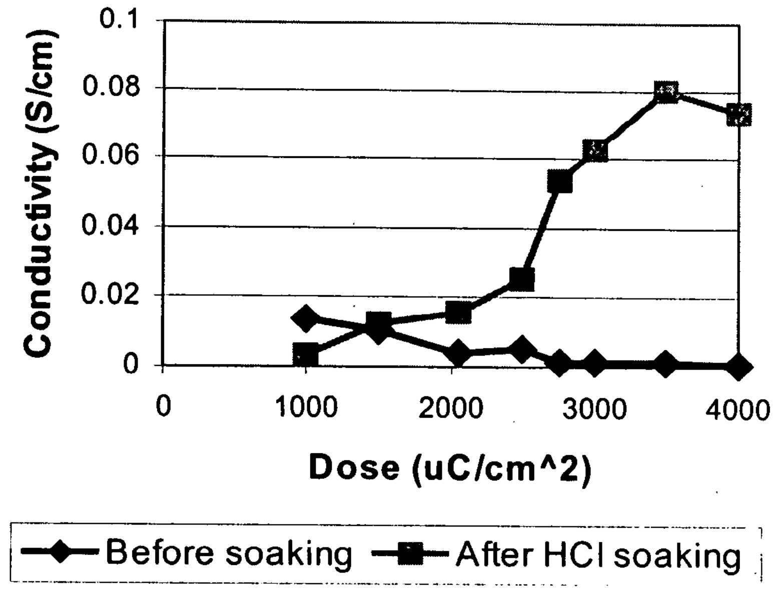


After crosslinking

# Conductivity of PPSA




**Unmodified**




**Doped with Diazide**

- Soaked in 10% HCl for 24 hours
  - Increased ionic conductivity
- 3 wt% diazide* added to PPSA
  - Promotes crosslinking, thus reducing necessary electron beam dose

*2, 6-bis(4-azidobenzylidene)-4-ethyl cyclohexanone

71

# Silicon Dioxide as a Membrane

- Plasma-Enhanced Chemical Vapor Deposition (PECVD) at 75-150°C
  - Temperature is shown to have a large effect on permittivity and loss*
    - Lower temperatures lead to increased silanol and water concentrations
    - The hydroxyl groups lead to increased polarity, and therefore higher ionic conductivity
- Support for polymer membranes
  - Mechanical support
  - Solvent Barrier
- Possible stand-alone membrane

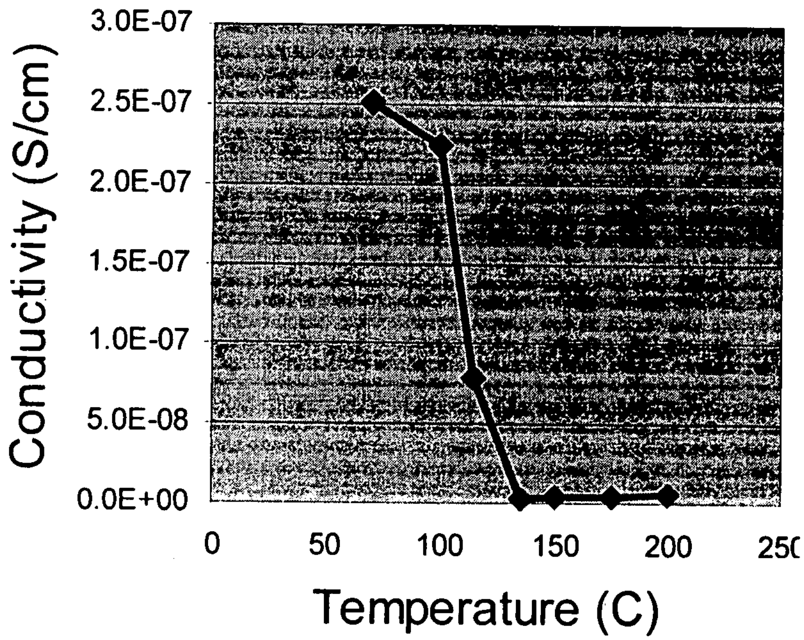
* Ceiler, Kohl, and Bidstrup, *J. Electrochemical Society*, Vol. 142, No. 6, pp. 2067-2071

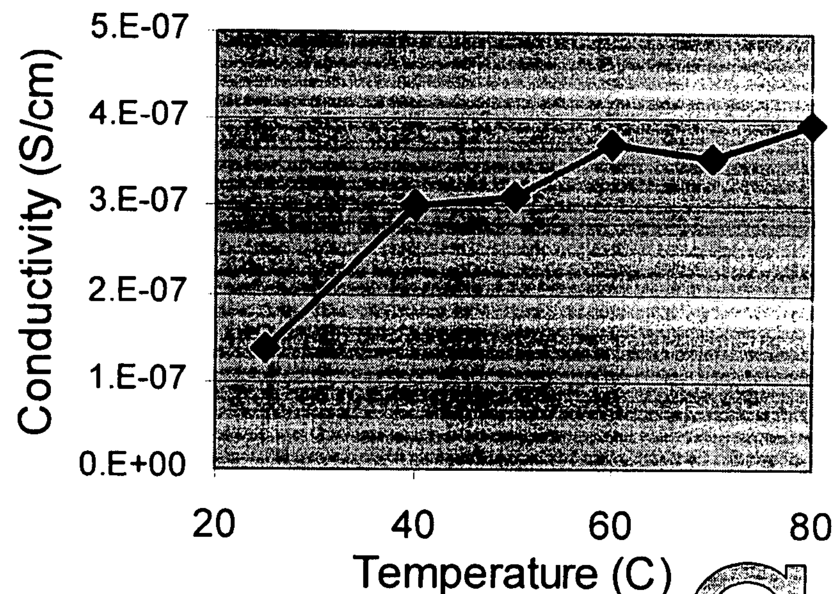
(13)

# Conductivity of Silicon Dioxide

- Increasing ionic conductivity of $SiO_2$ films with decreasing deposition temperature
- Measured through the use of impedance spectroscopy

- Increasing ionic conductivity of $SiO_2$ films with increase in temperature
- Measured through current step experiments

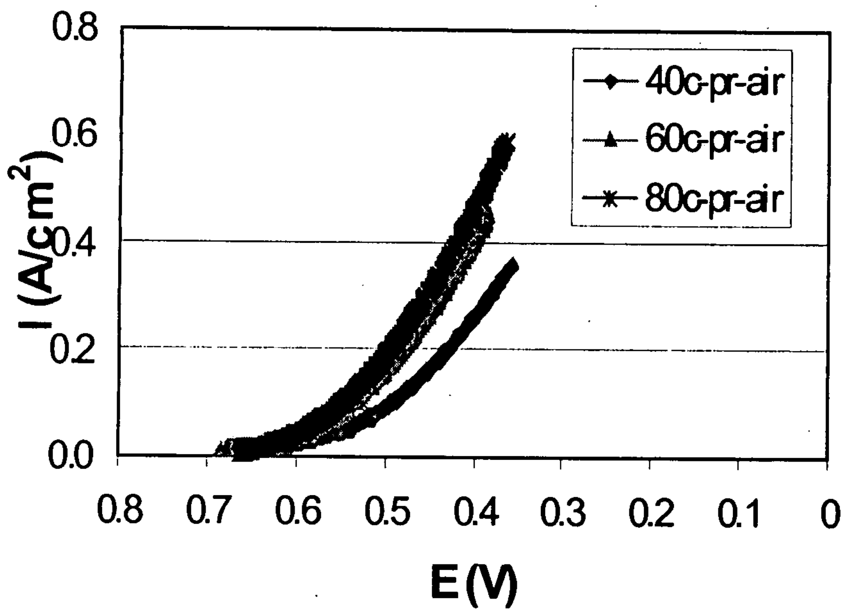


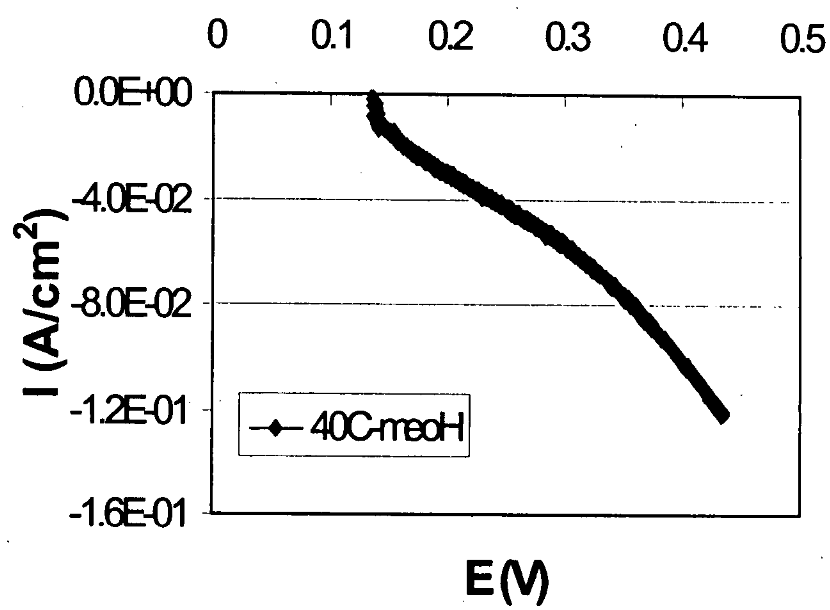
(14)

# $SiO_2$ Membrane Performance

## Pressurized Air Reduction

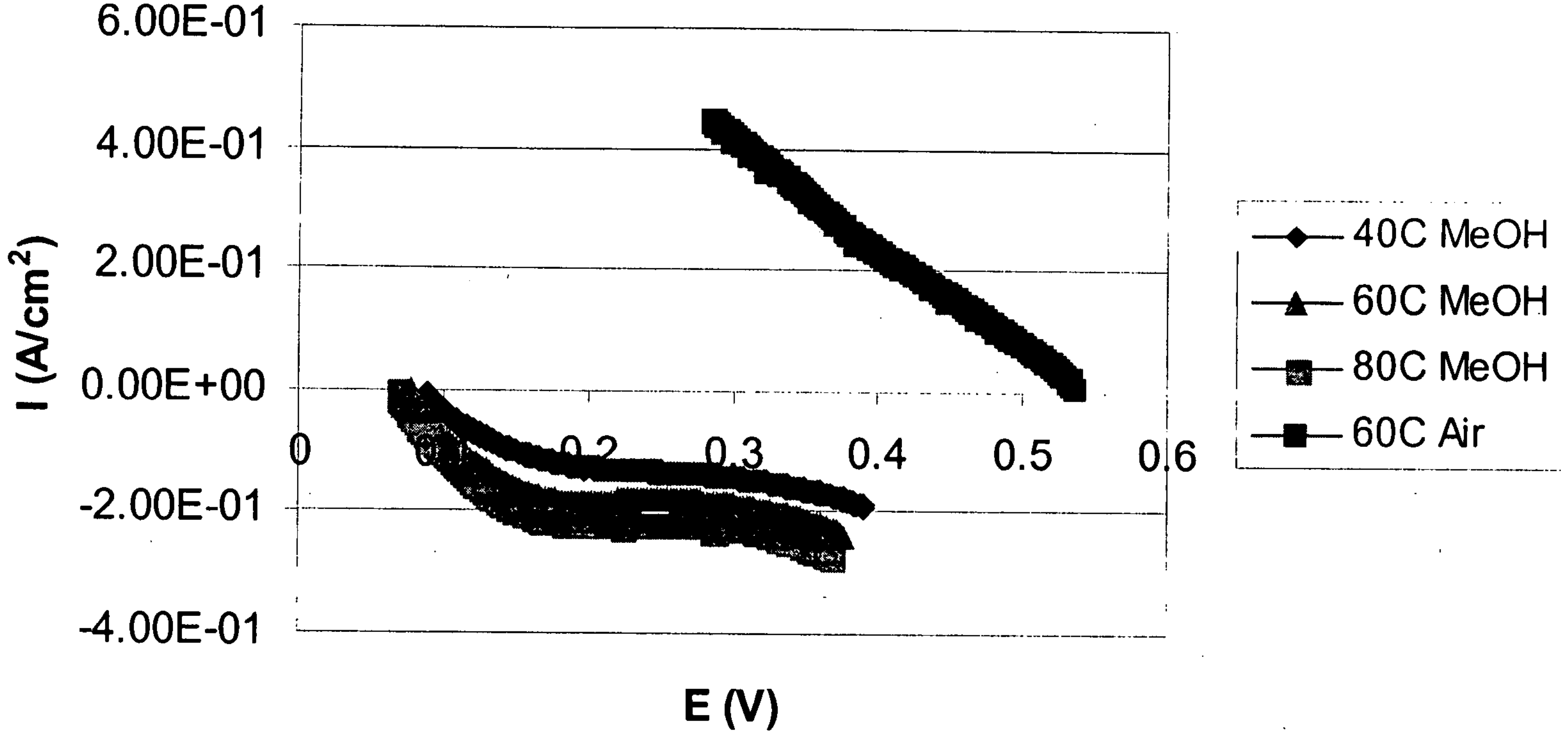



## Methanol Oxidation




$SiO_2$ thickness = 3.0 um
Preloaded Pt catalyst
Voltage vs. SCE
Scan rate = 1 mV/s

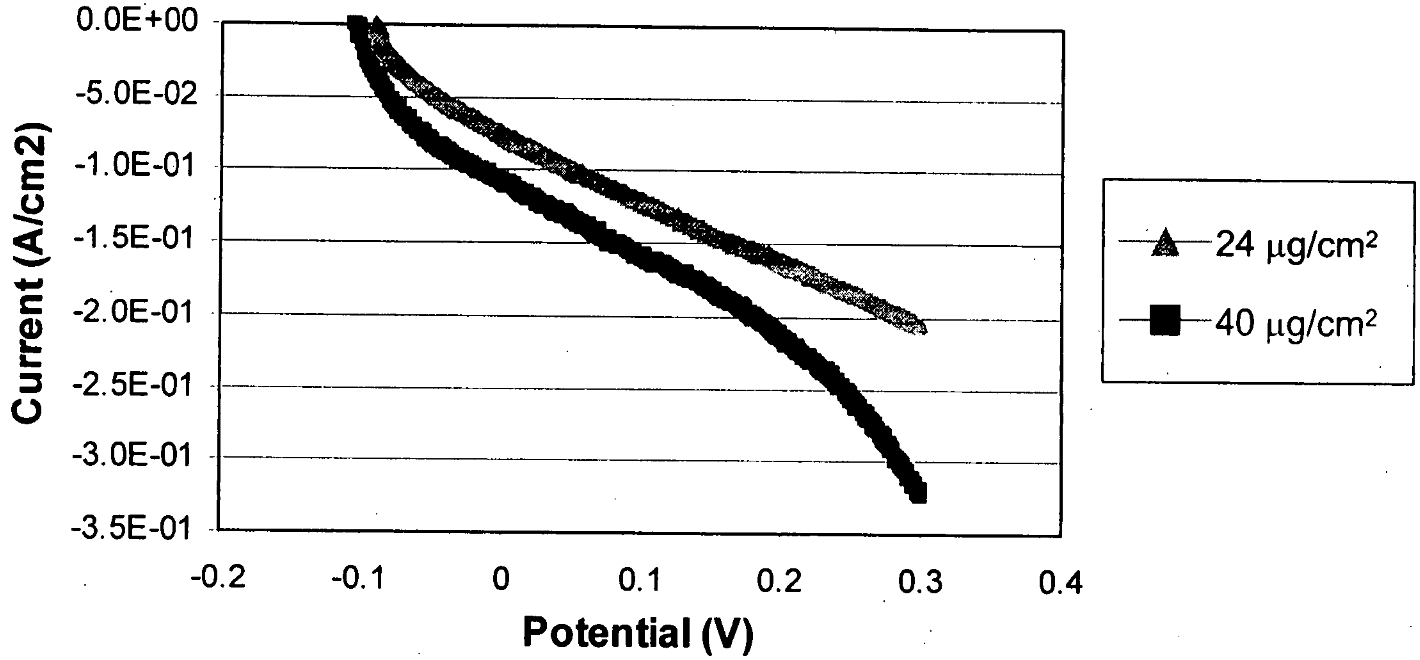
# $SiO_2$ Membrane Performance




$SiO_2$ thickness = 1.8 um
Preloaded Pt-Ru catalyst
Voltage vs. SCE
Scan rate = 1 mV/s

(15)

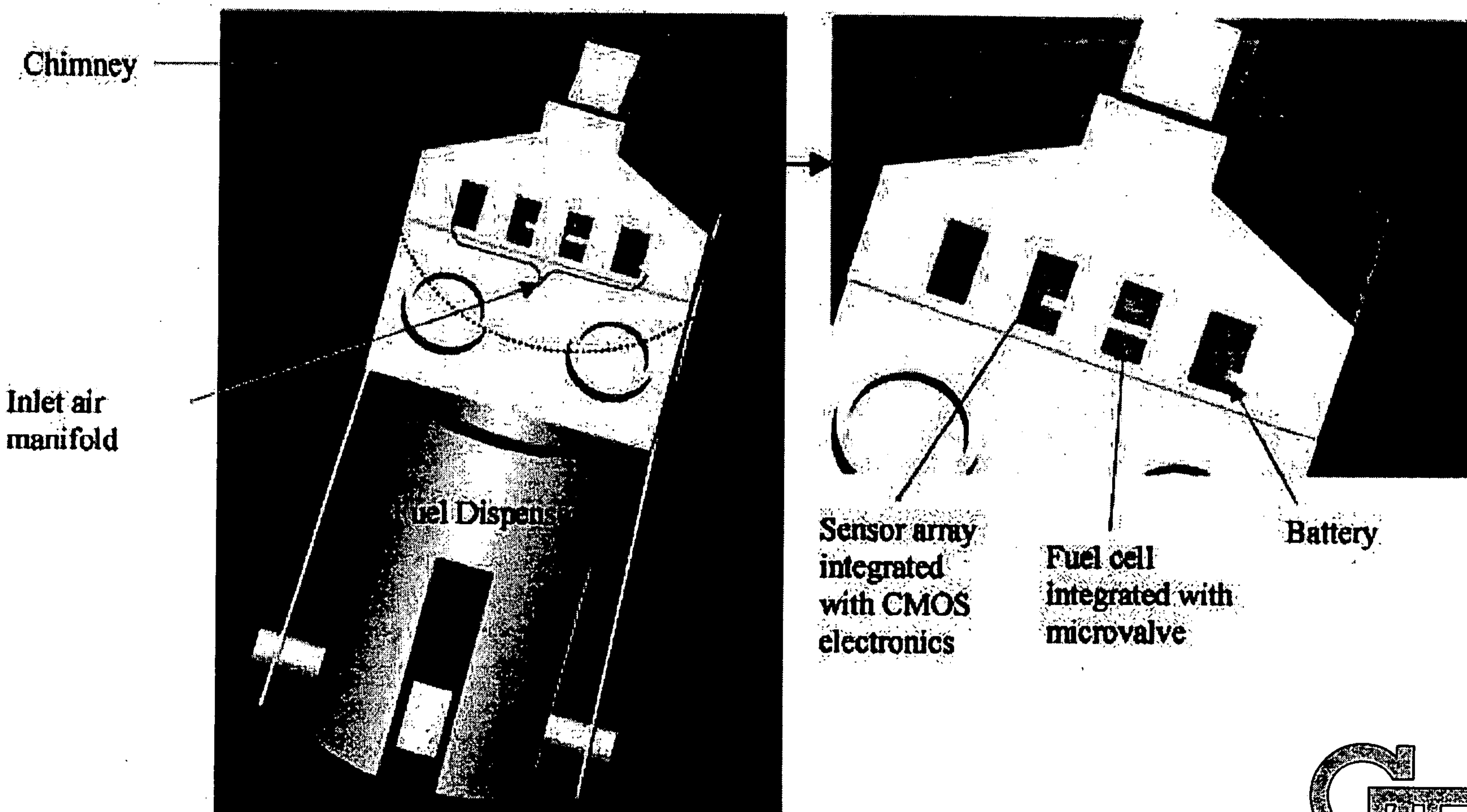
# Sputtered Catalyst Performance




Temperature = 60°C
$SiO_2$ thickness = 3.2 um
Sputtered Pt-Ru catalyst
Voltage vs. SCE
Scan rate = 1 mV/s

91

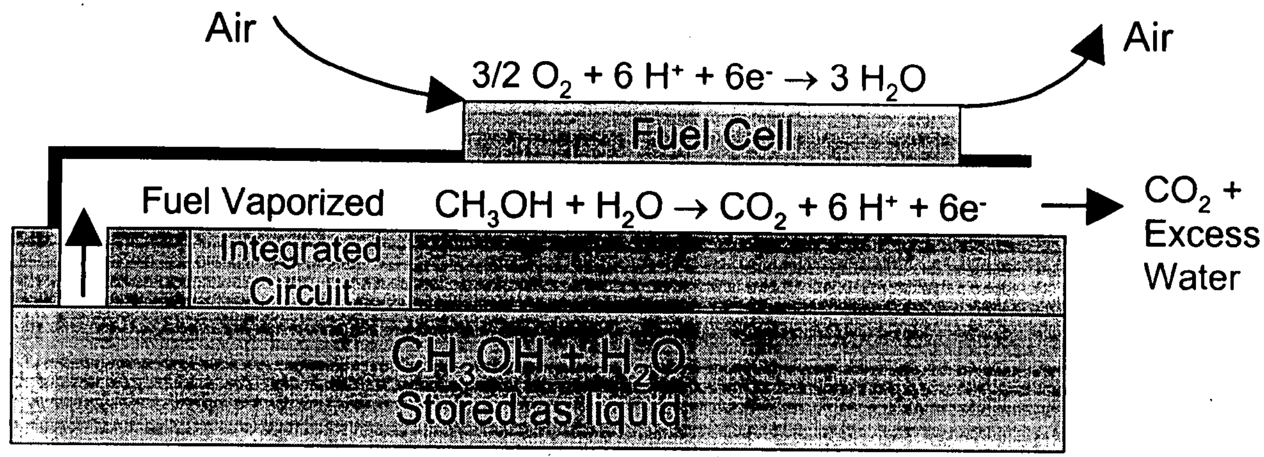
# Integrated Micro Fuel Cell/Si CMOS/Sensor Technology

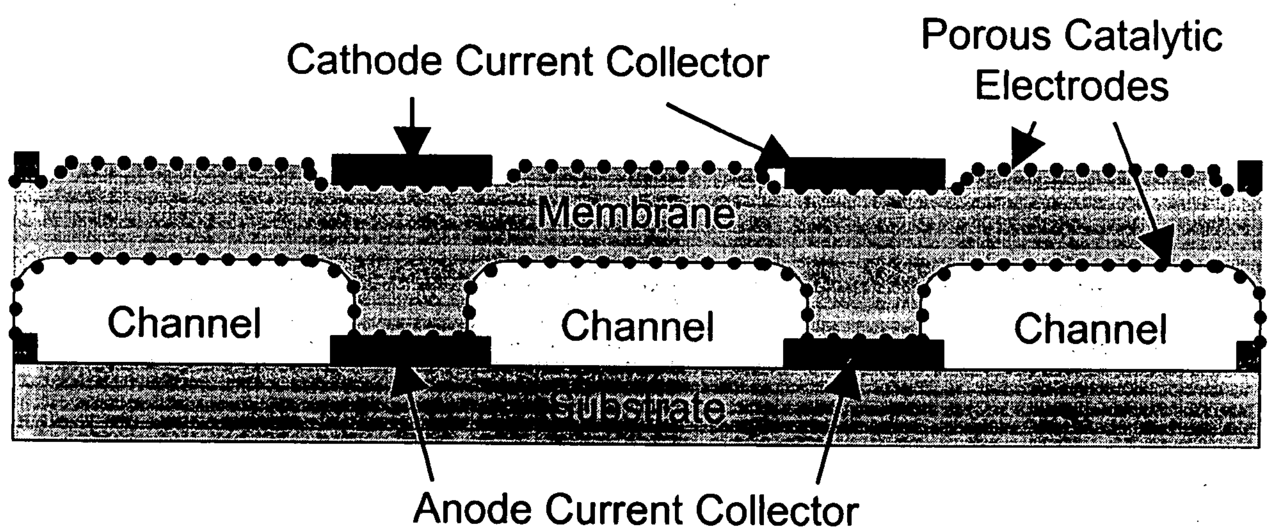



21

(81)

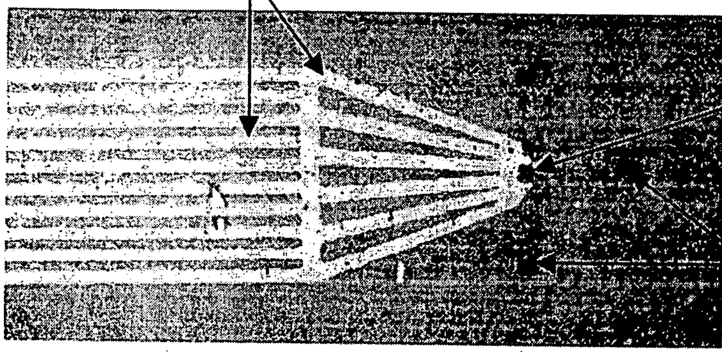
# Schematics of Micro Fuel Cell

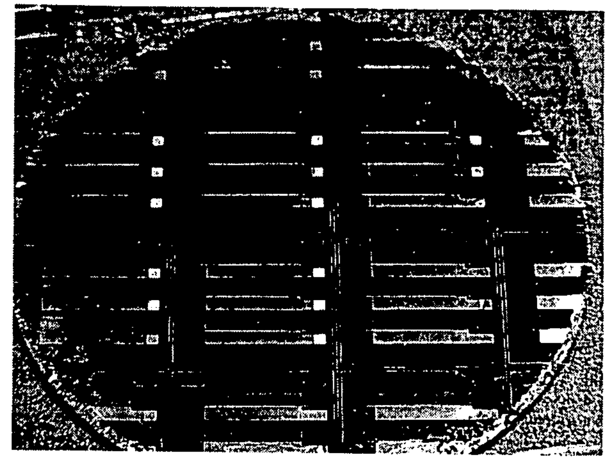


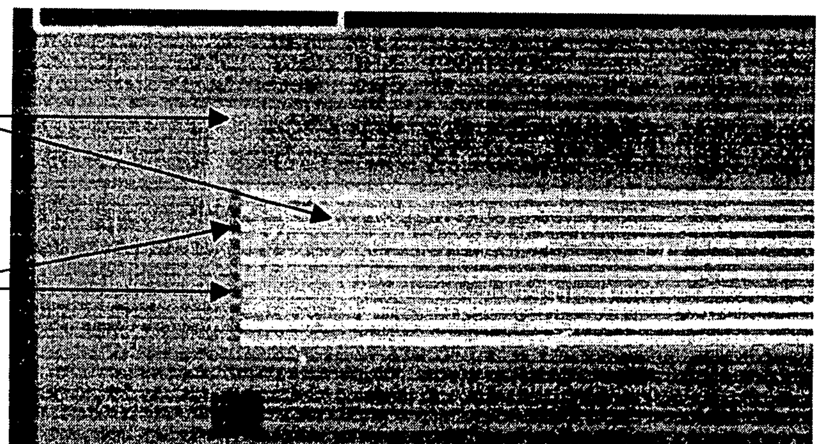



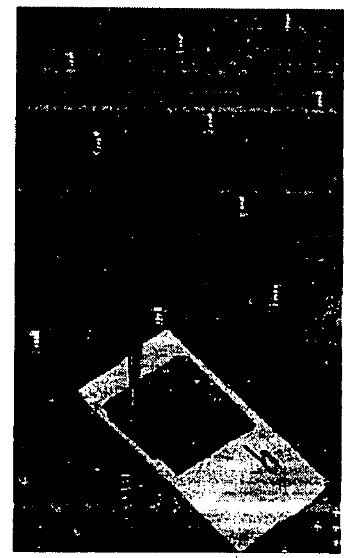
19

# Fabricated Prototypes





20

# Summary

- Thin-film membranes for use in proton exchange membrane (PEM) fuel cells have been developed.
  - Deposited through common microelectronic fabrication techniques, including spin-coating and PECVD
  - Incorporated into microfabricated fuel cell design
  - Needs mechanical support for larger designs
  - Low current density due to low catalyst loading

## Thin-film Membranes for Fuel Cells
## Disclosure Summary

Thin-film membranes for use in proton exchange membrane (PEM) fuel cells have been developed. Also included in the development are ways in which to support these films. These membranes are made through common micro-fabrication techniques, including spin-coating and plasma enhanced chemical vapor deposition (PECVD), and can have a thickness up to 20 micrometers. The materials include polymers, silicon dioxide, and doped silicon dioxide. The silicon dioxide membrane was deposited so as to have high ionic conductivity for proton exchange. The conditions for ionic conductivity were to have the deposition temperature low, such as 60°C to 200°C. PEM fuel cells membranes work by conducting protons from the cell anode to cathode. The thinner the membrane, the easier it is for protons to move through it, thus increasing the amount of electrical current that can be generated. Meanwhile, these thin-film materials are superior to currently used PEMs in preventing reactants from passing through the membrane, a common problem particularly in direct methanol fuel cells.

Slides 6 and 8 [illegible] shows electron beam crosslinking of a polymer proton exchange membrane. This is the first demonstration of the electron beam crosslinking.

One expected use of these thin-film membranes is in micro fabricated fuel cells. The films would be deposited directly onto the substrate during the fabrication sequence. One example of this is in a previous disclosure for micro fabricated fuel cells where the membrane is deposited onto a patterned sacrificial material, which when removed leaves microchannels for fuel flow.

In other applications, the delicate nature of such a thin material requires that it be supported. The support structures include anything with small holes or porous materials, such as fritted glass or a gas diffusion layer. A filler material, such as wax or polymer, is used to fill in the holes. It is then polished to expose the support structure. The membrane is then deposited on the smooth surface. Removal of the filler material leaves the supported membrane with exposed areas for contact between the membrane and reactants. Alternately, the use of a filler material with a high permeability to the reactants would not require its removal.

The fuel cell catalyst can be deposited with these membranes in a variety of methods, including through inks or sputtering. The deposition can be onto or into the support, in between the filler and membrane, or even imbedded into a non-removed filler.

Figure 1 shows a schematic diagram of a supported membrane in its testing setup. Figures 2 and 3 show the polarization curve for a 3-um thick $SiO_2$ film deposited onto a gas diffusion layer with a platinum catalyst loading of 0.35 mg/cm$^2$.

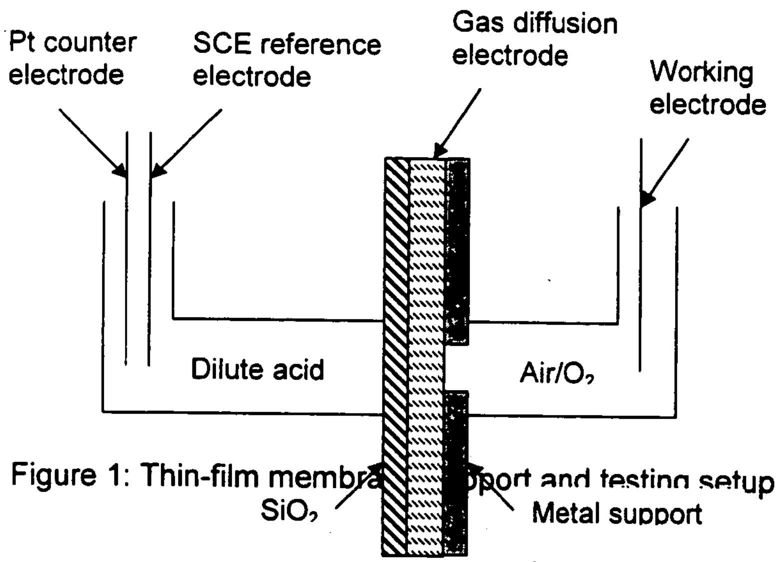



Figure 1: Thin-film membrane support and testing setup

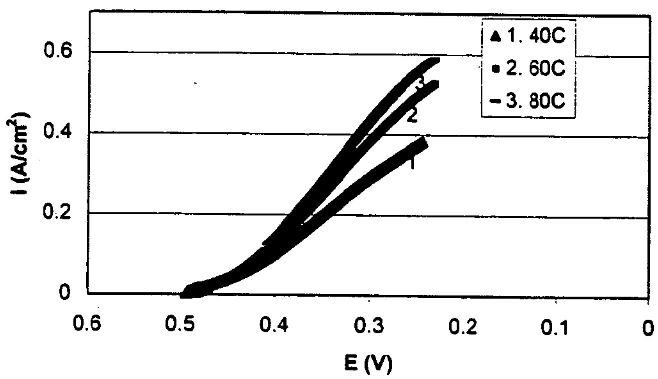



Figure 2: Cathode (air/$O_2$ half cell) polarization performance

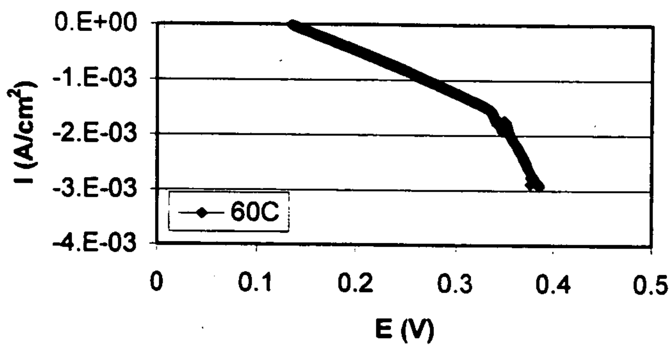



Figure 3: Anode (2M methanol) polarization performance

## Design Considerations

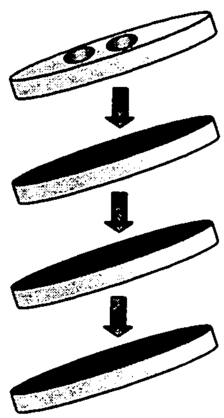
The following characteristics are desired for thin-film membranes and membrane-electrode assemblies.

Maximum exposure of membrane to fuel
Maximum activity of catalyst with low loading
High proton conductivity of membrane with no electrical conductivity
High electrical conductivity of current collector
Minimum methanol crossover through membrane, even with high concentrations of methanol feed

---

## Step-by-Step Fabrication

To fabricate and support a thin-film membrane on a catalytic gas diffusion layer:



Solid support structure with small holes for fuel/membrane contact. A mesh or porous structure may also be used.

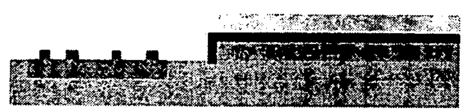
Cover the support with the gas diffusion layer (GDL).

If needed, the GDL can be coated with Nafion, or similar material, to fill in any uneven spaces.

Deposit thin film membrane through spin-coating or plasma enhanced chemical vapor deposition.

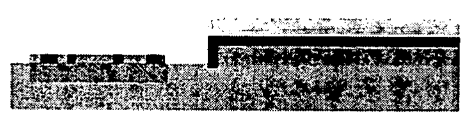
A process sequence for integrated micro fuel cells:

Fabricate Sensor and CMOS Devices



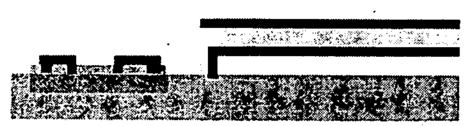
Overcoat Microchannels with Polymer Electrolyte Membrane

Pattern Sacrificial Polymer for Microchannels



Dielectric Cure and Decomposition of Sacrificial Polymer

First Metallization of CMOS and Anode Catalyst

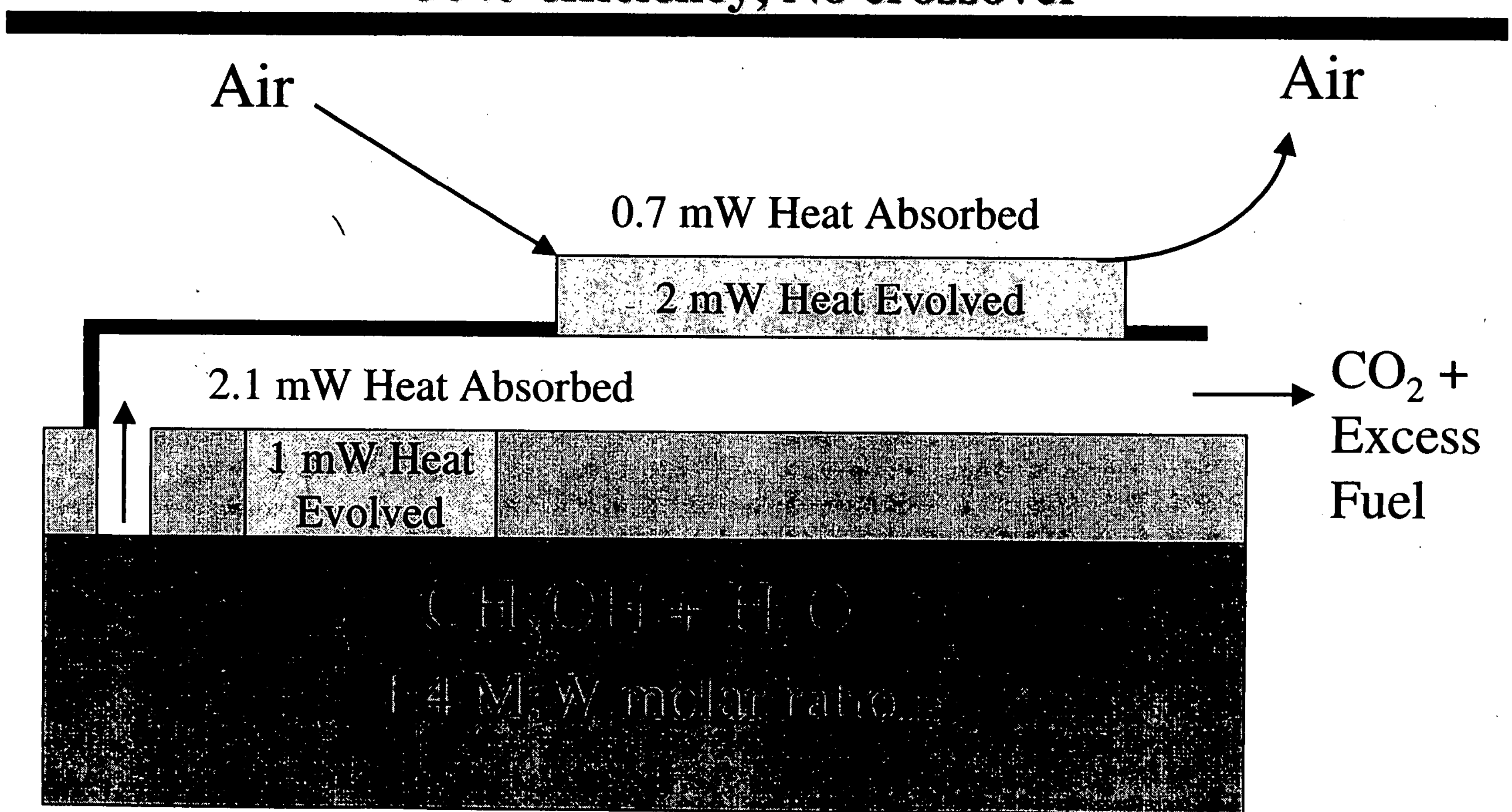


Second Metallization of CMOS and Cathode Catalyst

# Micro-Fuel Cell

**1 mW power at 0.4 V**

**50% efficiency, No crossover**




Georgia Institute of Technology

25